UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 8
1595 Wynkoop Street
DENVER, CO 80202-1129
Phone 800-227-8917
http://www.epa.gov/region08

Ref: 8EPR-SR December 20, 2007

MEMORANDUM

SUBJECT: Libby Asbestos Site, Operable Unit 3
Development of the Tree Bark Sample Preparation Method

FROM: Bonnie Lavelle
Remedial Project Manager

TO: Libby Operable Unit 3 Site File

The attached documents, consisting of email messages and photographs exchanged between EPA and its contractors, Remedium, and EMSL Analytical, Inc., provide background information on the development of the standard operating procedure SOP TREE-LIBBY-OU3 (Rev. 1), "Sampling and Analysis of Tree Bark for Asbestos", approved by EPA on November 20, 2007.

Enclosures

Color Photo(s)

The following pages contain color that does not appear in the scanned images.

To view the actual images, contact the Region VIII Records Center at (303) 312-6473.

EMSL ANALYTICAL, INC.
107 HADDON AVENUE
WESTMONT, NJ 08108
PHONE: (845) 469-8671
FAX: (845) 231-6017

Sample Preparation of Libby Tree Bark

Bark test samples received in zip-lock plastic bags

Initial Test Sample

Diameter: 46mm
Initial Weight: 12.19g
Dry weight: 10.19g

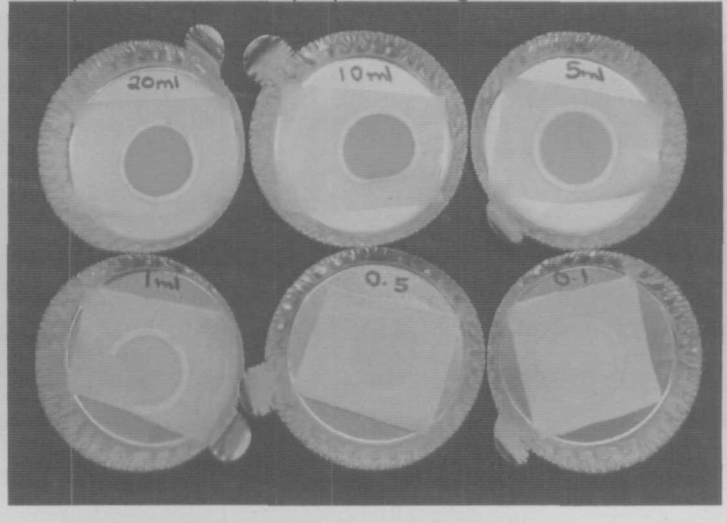
The initial trial involved an incremental ramp and soak scenario as follows:

200 C for 1 hour: no visible change in sample
300 C for 3 hours: smoking, core color now black
Smoking now minimal
450 C for 18 hours

Ashing Results

Sample ashed nicely.
Only 1.2% of the sample remains

- The ash was rinsed into a disposable beaker and brought up to 100ml with DI water
- Sample was sonicated for 30 minutes (sonicator calibrated to ASTM 5755 specs)
- Various filtrations were performed to find proper loading



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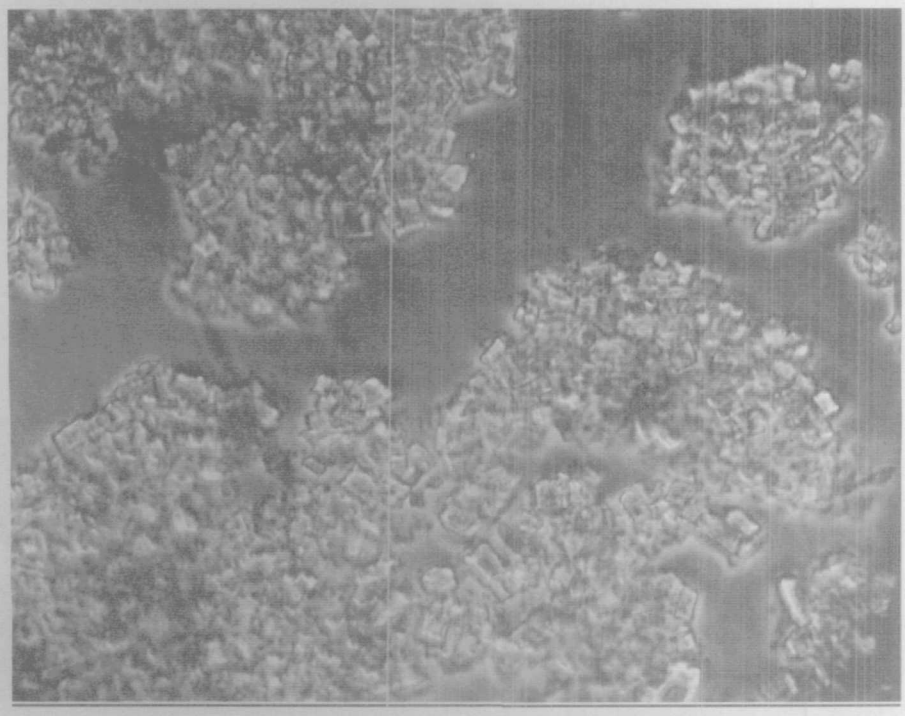
The 1 ml prep was dried and prepped for PCM to get an indicator of sample loading and appearance under light microscopy.



Follow Up

Two extra filtrations at 0.1 and 0.5 ml were performed, this time with the sample being diluted with 0.1 N HCL to test if this might reduce the clumping of residue seen above.

Pictures when available

Going forward (action items)

1. Streamline the ashing process by reducing the ramp to 450 C
2. HCl or no HCl?
3. find optimum dilution
4. test run analysis including use of the EDD spreadsheet

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Going forward

Actual samples (not test samples) were received at the lab.

Many were of quite different makeup than the test samples.

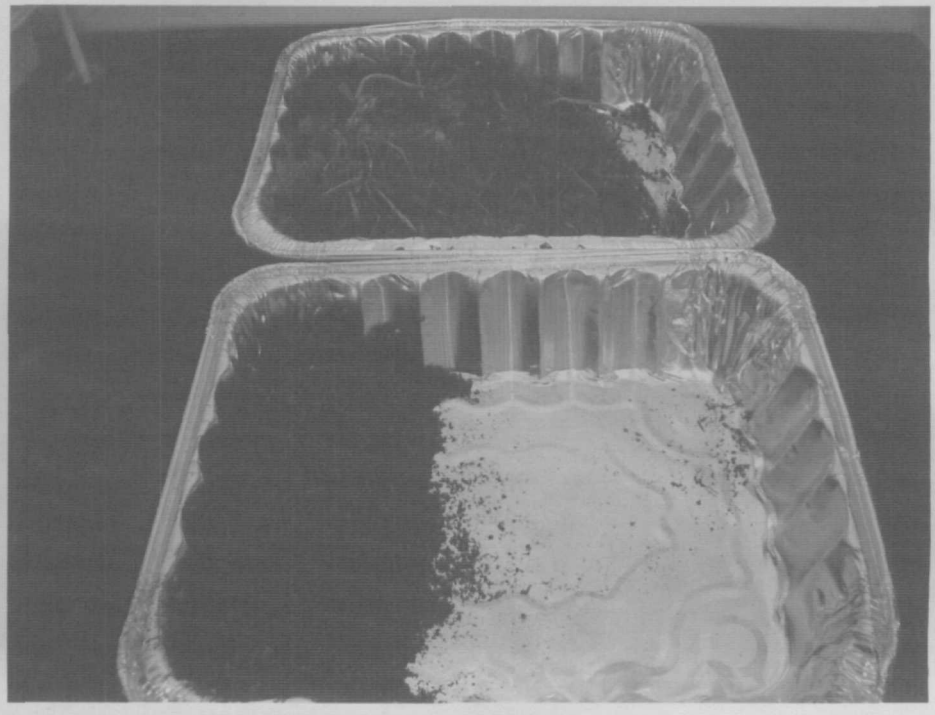
Many were fairly wet and with a fair amount of soil included.

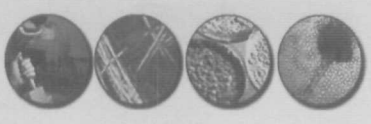
This is sample P1-00089
Station ID: SL 135-06

To give everyone a feel for the volume of the soil component the sample was dried and sieved through a 2 mm sieve.

The top pan contains the > 2mm fraction (471.87g)

The bottom pan contains the < 2mm fraction (232.47)





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Sample Preparation of Libby Duff

Duff test samples received in zip-lock plastic bags

Sample 1

Initial Weight: 184.97g
Dry weight: 174.29g

The initial trial involved an incremental ramp and soak scenario as follows:

100 C for 1 hour:	no visible change in sample
200 C for 1 hour:	no visible change
300 C for 3 hours:	smoking, core color now black
400 C for 2 hours	smoking stopped, sample color black
450 C for 18 hours:	

Results

Approximately 50% reduction in both volume and mass.

Some complete ashing but much of the sample is still intact and black (not grey)

"Bill Brattin"
<brattin@syrres.com>
10/10/2007 05:03 PM

To Dan Wall/EPR/R8/USEPA/US@EPA
cc Bonita Lavelle/EPR/R8/USEPA/US@EPA
bcc
Subject RE: Fw: Libby OU3 tree bark and organic debris

What is SHMP?

sodium hexametaphosphate. We have tentatively decided it should not be used because of the potential to increase the Na content of fibers. EMSL is currently testing to see if H2O alone or else weak acid can dissolve the residue without adding an ionic constituent.

Do they measure thickness of bark?

No, but they measure diameter and weight. Original plan to saw off back is now fropped, since the sample is really not very thick.

How do they propose to homogenize the duff? Can they grind it without loss of fiber similar to soil?

Not resolved yet. This is the most difficult problem. Several ideas are being floated.

What about an acid digestion of the duff?

Possible, but this would likely alter the metal content of the fibers which would be undesirable

Is smoking a problem for the lab personnel or is it a loss of fibers or both?
Don't know. They are responsible for their own safety. We only need to ask about the potential for fiber loss.

Why did they have a more agressive temperature ramp for the duff?

Larger mass. It took 18 hrs and was only partially ashed at that time

Why and what do they soak the samples in?

I think "soak" is jargon for holding at a constant temp.

What exactly is in the duff? leaves needles bark twigs?

Yes, all of the above, plus some dirt

****

Bill Brattin
Syracuse Research Corporation
999 18th Street Suite 1975
Denver CO 80202
Phone: 303-357-3121
Fax: 303-292-4755
e-mail: brattin@syrres.com

-----Original Message-----
From: Wall.Dan@epamail.epa.gov [mailto:Wall.Dan@epamail.epa.gov]
Sent: Wednesday, October 10, 2007 4:52 PM
To: Lavelle.Bonita@epamail.epa.gov
Cc: brattin@syrres.com
Subject: Re: Fw: Libby OU3 tree bark and organic debris

Bonnie/Bill
Some questions when you get time. I am heading out in a few minutes so maybe we talk tomorrow or type something out if you want.

What is SHMP?
Do they measure thickness of bark?
How do they propose to homogenize the duff? Can they grind it without loss of fiber similar to soil?
What about an acid digestion of the duff?
Is smoking a problem for the lab personnel or is it a loss of fibers or both?
Why did they have a more agressive temperature ramp for the duff?
Why and what do they soak the samples in?
What exactly is in the duff? leaves needles bark twigs?

Dan

Bonita Lavelle/EPR/R8/USEPA/US
10/10/2007 04:24 PM

To: Dan Wall/EPR/R8/USEPA/US@EPA
cc:
Subject: Fw: Libby OU3 tree bark and organic debris

Dan

here's some info on the tree bark and duff prep.

they had a conference call this afternoon - I was not available. I had suggested tomorrow but due to the urgency, Bill Brattin participated and briefed me a little on what's going on. I'll call you....

----- Forwarded by Bonita Lavelle/EPR/R8/USEPA/US on 10/10/2007 04:20 PM -----

"Cahill, Ed" <ECahill@EMSL.com>

To

10/10/2007 09:44 AM	Bonita Lavelle/EPR/R8/USEPA/US@EPA, <brattin@syrres.com>, <Robert.R.Marriam@grace.com>, <brattin@syrres.com>, "Lynn Woodbury" <woodbury@syrres.com>	
	cc	
	"DeMalo, Robert" <RDemalo@EMSL.com>, "Denton, Robyn" <rdenton@EMSL.com>, "LaCerra, Charles" <CLaCerra@EMSL.com>	
	Subject	
	RE: Libby OU3 tree bark and organic debris	

To All:

We are actively working on the prep procedure for both the bark and duff samples. Observations so far:

Tree Bark
- cores were of a more handleable size than feared. And quite consistent in size. 46 mmm seems to be the magic number. Going forward we plan on doing these measurements with a caliper, measuring in 2 directions at 90 degrees to each other.
- to minimize possible fiber loss by excessive handling we decided to try ashing the whole core intact.
- we did do a ramp and soak scenario, starting at 100 C and increasing by 100 degrees each hour to get up to 450 -at about 300 degrees we were getting a pretty smokey but everything seems workable. I am fairly confident that we can shorten the ramp and soak time going forward.
-with 18 hours at 450 C the core is completely ashed. Only 1.2% by weight remaining -still remains to be seen what portion of that ash passes through the filter (we'll be attempting that shortly) (without the SHMP)

Duff
-here the sample size was larger than we were expecting at the lab.
- we attempted the most optimistic scenario, attempting to ash the entire sample.
-again a ramp and soak scenario starting at 200 C and increasing by 100 degrees each hour till we reached 450 C
- smoking was a definite problem.
- with 18 hours at 450 C the result was less than optimal. There was a large component that was still intact.

Tree Bark Ash Duff Ash
(Embedded image moved to file: pic31251.gif)(Embedded image moved to file: pic22073.gif)

My thought at this stage is to try ashing the duff in multiple crucibles (it would take quite a few) in order to

increase the ashing efficiency.
But at this point we would like to know your thoughts on how important it is to prep the entire sample.
Maybe we can all get together on a call this afternoon? Rob DeMalo and I are available if you wish.
By 2pm EST we should have something to report on the filtering process.

Ed Cahill
845-469-8671 (cell)

-----Original Message-----
From: Lavelle.Bonita@epamail.epa.gov
[mailto:Lavelle.Bonita@epamail.epa.gov]
Sent: Tuesday, October 09, 2007 7:47 PM
To: DeMalo, Robert; LaCerra, Charles
Cc: brattin@syrres.com; Robert.R.Marriam@grace.com; robert.j.medler@grace.com; Robert.R.Marriam@grace.com
Subject: Libby OU3 tree bark and organic debris

Dear Rob and Charlie:

Since most of the tree bark and organic debris sampling in the vicinity of the Libby mine will be finished this week, we're anxious to hear about your progress on the tree bark and organic debris (duff) sample preparation methods.

Are you available this week to join us in a conference call to let us know how things are progressing?

Thursday or Friday afternoon this week would work -

Here's some of the information we'd like to discuss - I know you've been in contact with the folks at SRC about some of this, but it might be beneficial to review it for us all:

A: Tree bark
1) What is the average diameter of the tree bark samples?
2) Are you still of the opinion that it's not useful to slice the tree bark plug horizontally to remove the back layer from the exposed surface layer? (we had discussed this as an option for reducing the mass that must be ashed)
3) What is the best sequence of temperature ramping (temps and holding times) for ashing the bark sample ?
4) Have you tried suspending the ashed residue in FDI? Results?
5) What is the recommended sonication setting (energy level)?

B: Duff
1) Is is possible and/or desirable to attempt to "homogenize" the duff sample?
2) What is the best way to ash the sample?

Please let me know if a conference call is possible. Thanks very much.

Sincerely,

Bonnie Lavelle
Remedial Project Manager

Libby Asbestos Superfund Site, OU3
EPA Region 8
1595 Wynkoop Street
8EPR-SR
Denver, CO 80202-1129

(303) 312-6579
Fax (303) 312-7151

"Lynn Woodbury"
<woodbury@syrres.com>
10/16/2007 10:23 AM
Please respond to
<woodbury@syrres.com>

To "'Bill Brattin'" <brattin@syrres.com>
cc Dan Wall/EPR/R8/USEPA/US@EPA, Bonita Lavelle/EPR/R8/USEPA/US@EPA
bcc
Subject FW: Bark and Duff Prep Summary to Date

History: This message has been forwarded.

Key notes from today's call w/ EMSL:

TREE BARK
--0.1N HCl worked great in dissolving crystalline residues, looks like filtered aliquot will be higher than 0.5 mL (probably 1-5 mL)
--Did not attempt heating procedure since acid worked
--Will perform round 2 of the tree bark prep to ensure that the procedure is reproducible
--Ed will update SOP (after he returns to the office) post-round 2
--ETA on updated tree bark SOP is mid next week
--I will follow up with field team to double-check sample inner diameter (1.75 inches vs. 1.85 inches) and establish the default sample area

DUFF - TEST SAMPLE
--initial ashing yielded 44% reduction, subsequent ashing resulted in only 2% additional reduction
--ashed sample is still pretty heterogeneous and doesn't result in a uniform filterable material
--will attempt freezer mill, mortar/pestle, acid to improve homogeneity and filterability
--potential drawback to physical alteration techniques is that the particle size distribution in duff may be altered
--ETA on when next round of prep results will be available in about one week

DUFF - REAL SAMPLE
--2 mm sieving yields 470 g duff sub-sample (>2 mm) 230 g "soil" sub-sample (<2 mm)
--ashing of < 2 mm sub-sample yields only an 18% reduction - which confirms that this sub-sample is primarily soil
--one option is to send the < 2 mm sub-sample back to Troy Prep Lab for PLM-VE prep
    (note: preference would be to keep this sub-sample separate from the 0-2" forest soil sample to avoid potential dilution)
--discussion was tabled until filterability results from test sample are available

-----Original Message-----
From: Ed Cahill [mailto:ecahill@emsl.com]
Sent: Tuesday, October 16, 2007 8:45 AM
To: 'Bill Brattin'
Cc: 'Robert DeMalo'; 'Charles LaCerra'; 'Robyn Denton'; 'Bonnie Lavelle'; 'Dan Wall'; 'Marriam, Robert R.'; 'Lynn'
Subject: RE: Bark and Duff Prep Summary to Date

Re: 'todays meeting see attached

Ed Cahill
National Director Asbestos Services

EMSL Analytical, Inc.
phone: 845-469-8671 mobile: 845-238-4559 fax: 845-231-6017

-----Original Message-----
From: Bill Brattin [mailto:brattin@syrres.com]
Sent: Tuesday, October 16, 2007 9:25 AM
To: 'Ed Cahill'
Cc: 'Robert DeMalo'; 'Charles LaCerra'; 'Robyn Denton'; 'Bonnie Lavelle'; 'Dan Wall'; 'Marriam, Robert R.'; 'Lynn'
Subject: RE: Bark and Duff Prep Summary to Date

ok, lets plan on a call at 11:00 EST (9:00 MST).

Call in number = 866-299-3188 Code = 303-312-6579

****
Bill Brattin
Syracuse Research Corporation
999 18th Street Suite 1975
Denver CO 80202
Phone: 303-357-3121
Fax: 303-292-4755
e-mail: brattin@syrres.com

-----Original Message-----
From: Ed Cahill [mailto:ecahill@emsl.com]
Sent: Tuesday, October 16, 2007 7:03 AM
To: 'Bill Brattin'
Cc: 'Robert DeMalo'; 'Charles LaCerra'; 'Robyn Denton'; 'Bonnie Lavelle'; 'Dan Wall'; 'Marriam, Robert R.'; 'Lynn'
Subject: RE: Bark and Duff Prep Summary to Date

I need to head out around noon so 11 am will be fine.

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Ed et al.

Based on your e-mail, I am assuming that we should try to have a call this morning.

I have a meeting from 8:30 to about 10:00 in the AM, but Lynn could sit in for me if that works for others.

Bonnie?

****

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e-mail: brattin@syrres.com

-----Original Message-----
From: Ed Cahill [mailto:ecahill@emsl.com]
Sent: Friday, October 12, 2007 7:14 PM
To: 'Bill Brattin'
Cc: 'Robert DeMalo'; 'Charles LaCerra'; 'Robyn Denton'; 'Bonnie Lavelle'; 'Dan Wall'; 'Marriam, Robert R.'
Subject: RE: Bark and Duff Prep Summary to Date

Hey Bill:
1) understood
2) will do. Should be able to do more than 5g per, we'll see. I could see even by eye that a large portion of the volume of the sub 2 mm fraction is organic. By mass I'm sure the mineral component is much higher. At this point we won't get them in the oven till Monday a.m.
I can be available Tuesday a.m. but I'm flying in the afternoon.

I'll be back in town on Monday the 22nd but you can always reach me on the cell.

Ed
phone: 845-469-8671 mobile: 845-238-4559
-----Original Message-----
From: Bill Brattin [mailto:brattin@syrres.com]
Sent: Friday, October 12, 2007 8:05 PM
To: 'Ed Cahill'
Cc: 'Robert DeMalo'; 'Charles LaCerra'; 'Robyn Denton'; Bonnie Lavelle; Dan Wall; 'Marriam, Robert R.'
Subject: RE: Bark and Duff Prep Summary to Date

Ed
This is pretty interesting. Based on a recent discussion I have had with EPA, I would like to suggest the following:
1) As we discussed, please keep your major attention on tree bark, with solving our duff problems in the background
2) Howver, with regard to duff, we are wondering if you could take a reasonable size sample (maybe 5 grams or so?) of the "soil" (i.e., the sub-2 mm fraction) and the "duff" (i.e., did not pass the 2 mm screen), and try ashing them in crucibles. The purpose is to see how much by mass of each fraction is organic and how much is mineral.
If possible, we are hoping you could do that on Monday, and then be able to relay all findings on tree bark and duff in a call on Tuesday.

Please let me or Bonnie know if that seems reasonable.

Thanks

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Fax: 303-292-4755

e-mail: brattin@syrres.com

-----Original Message-----
From: Ed Cahill [mailto:ecahill@emsl.com]
Sent: Thursday, October 11, 2007 10:26 PM
To: 'Marriam, Robert R.'; 'Bill Brattin'; woodbury@syrres.com; 'Bonnie Lavelle'
Cc: Robert DeMalo; Charles LaCerra; Robyn Denton
Subject: Bark and Duff Prep Summary to Date

Attached are summaries of progress to date on the sample prep for both the bark and duff.
As we hone the procedures for the bark samples I'll incorporate our findings into the existing SOP.

We are trying subdividing a duff sample into smaller vessels during ashing to see if we can get complete ashing that way.

I'll keep everyone up to date as data comes in.
If anyone has suggestions or ideas don't hesitate to contact me.
Thanks

Ed Cahill
National Director Asbestos Services
EMSL Analytical, Inc.
phone: 845-469-8671 mobile: 845-238-4559 fax: 845-231-6017


2007 10 16 duff.pdf


2007 10 16 tree bark.pdf

"Ed Cahill"
<ecahill@emsl.com>
10/22/2007 07:51 AM

To "'Ed Cahill'" <ecahill@emsl.com>, "'Bill Brattin'" <brattin@syrres.com>
cc Bonita Lavelle/EPR/R8/USEPA/US@EPA
bcc
Subject RE: Bark and Duff Prep Summary to Date

sorry
I hit the send button prematurely

From: Ed Cahill [mailto:ecahill@emsl.com]
Sent: Monday, October 22, 2007 9:45 AM
To: 'Bill Brattin'
Cc: 'Bonnie Lavelle'
Subject: RE: Bark and Duff Prep Summary to Date

Hi Bill

Glad to hear that use of acid helps break up the crystaline residue from tree bark ashing.
Maybe you have this all figured out already, but here are my thoughts:

1) The residue should be resuspended and sonicated in weak acid, rather than being suspended in water, aplied to the filter, and then rinsed with acid. The latter would result in uneven distribution of material on the filter.
Absolutely

2) The strength of the acid should not be any higher than needed to get good dissolution of the soluble portion of the residue. For example, maybe you begin by dissolving/suspending the residue in 0.1 N HCl, but as soon as dissolution has occurred, dilute the suspension with DFI water to yield a lower conce (e.g., 0.01 N HCL). The reason this is important is that we do not want to alter the mineral content of the fibers any more than we have to.
Understood. I understand where you are coming from however 0.1 N is quite dilute. As I have said the NY ELAP method (Jim Webber's) uses concentrated HCl! I think more important is the time exposed to the acid. Over time some leeching may start to occur. Robyn has some filters to prep today. If the salts are not present I would propose shortening the sonication time from 30 to 15 minutes.

****

Bill Brattin
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Attached are summaries of progress to date on the sample prep for both the bark and duff.
As we hone the procedures for the bark samples I'll incorporate our findings into the existing SOP.

We are trying subdividing a duff sample into smaller vessels during ashing to see if we can get complete ashing that way.

I'll keep everyone up to date as data comes in.
If anyone has suggestions or ideas don't hesitate to contact me.
Thanks

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